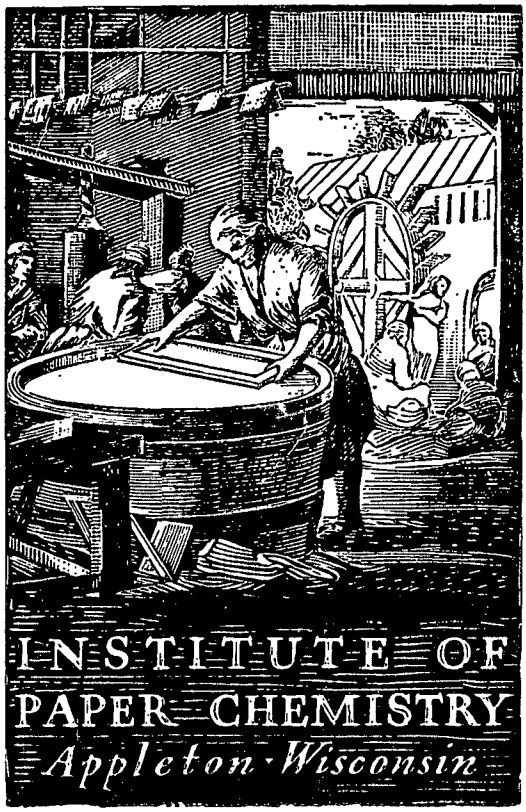

COMPRESSION FAILURE MORPHOLOGY OF LINERBOARD

Project 2695-20

Report One
A Progress Report
to
FOURDRINIER KRAFT BOARD GROUP
of The
AMERICAN PAPER INSTITUTE

November 1, 1978

THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

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SUMMARY

The importance of compressive strength in corrugated box end use performance is widely recognized. One common compression failure occurs from top to bottom loading of corrugated boxes during warehouse stacking. Recent research at The Institute of Paper Chemistry indicates that the compressive strength of the liners are a key quantity in the compressive strength of combined board. Unfortunately, very little is known about the sequence of events that take place within the liner during compression failure. As a first step in a program to define the important parameters in compression failure of linerboard, it was hypothesize that the fiber to fiber bond strength and the fiber stiffness are the dominant factors in controlling the compression failure of linerboard. The purpose of this project is to prove or disprove the hypothesis.

Earlier work at The Institute of Paper Chemistry suggested that the compression failure of linerboard is initiated by at least two different mechanisms, these two mechanisms becoming apparent when a plot of the compressive strength of the linerboard is developed against the z-direction tensile strength of the liner. The z-direction tensile strength is an indication of fiber to fiber bond strength. An increase in bond strength for a weakly bonded liner increases the compressive strength substantially; however, an increase in bond strength of a relatively well bonded liner has little effect on the compressive strength of the liner. Photomicrographs of the edge of the failed liners appear to confirm the hypothesis

of two distinct mechanisms. Some liners show a delamination of the fiber mat which would be caused by a failure in the fiber to fiber bond (Fig. 1).

Figure 1. Bond Breakage

Photomicrographs of other liners that have failed due to a compressive load show regions within the fiber mat where the fibers appeared to have buckled (Fig. 2).

Figure 2. Fiber Buckling

Photomicrographs of still other liners show both a delamination of the fiber mat and fiber buckling (Fig. 3).

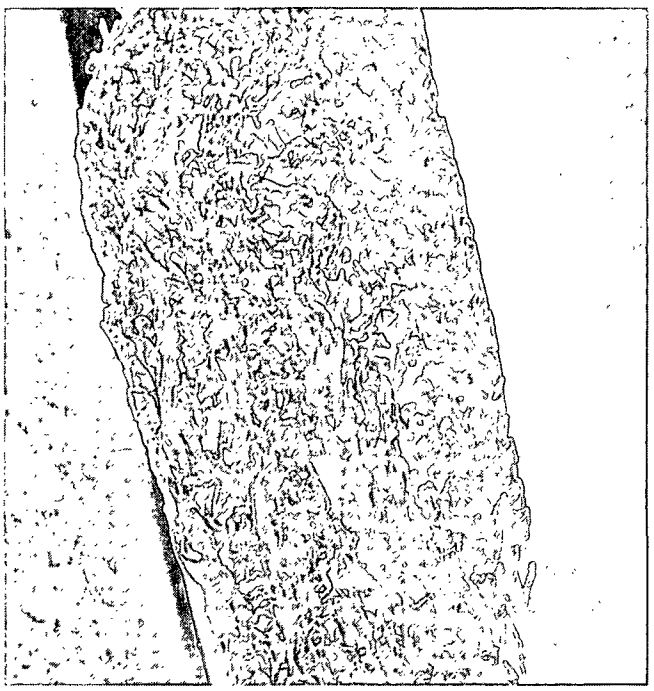

Figure 3. Fiber Buckling and Bond Breakage

The program developed for the determination of the role of fiber stiffness and bond strength in compression failure calls for the development of four sets of handsheets having predetermined physical properties, one set of handsheets having high bond density and high fiber stiffness, one set having low bond density and low fiber stiffness, one set having high bond density and a low fiber stiffness, and one set having low bond density and high fiber stiffness. Bond density is the bond force per relative bonded area. Bond force is measured by the TAPPI z-directional tensile test, and relative bonded area is measured by optical methods using a Beckman spectrophotometer. Fiber stiffness is the product of the moment of inertia of the fiber times its modulus of elasticity. For the purpose of this phase of the program it is assumed that the zero-span tensile strength is an indirect measure of fiber stiffness. Thus the necessary data can be developed to prove or disprove the hypothesis that the fiber bond and fiber stiffness are the key controlling factors in the compression failure of linerboard.

The handsheets prepared in this study were evaluated for ultimate compressive strength, compressive modulus of elasticity, relative bonded area, zero-span tensile strength, z-direction tensile strength, caliper, and basis weight. Photomicrographs of the compression failure of the sheets were obtained.

The development of the four sets of handsheets with the necessary properties has been only partially successful. The method used to vary the bond density of the handsheets was to change the wet pressing pressure from essentially zero up to 500 psi. The bond force of the handsheets, as measured by the TAPPI z-directional tensile test, increased with increasing wet pressing pressure. The bonded area also increased. The results of testing two sets of handsheets with varying amounts of wet pressing pressure showed the bond density increased for one set of handsheets and showed no increase in bond density for the other set. Further work in varying bond density needs to be done.

Earlier work at The Institute of Paper Chemistry shows a 60-70% increase in the tensile modulus of handsheets made from annealed fibers; therefore, annealing the pulp fibers appeared to be a practical means of varying fiber stiffness. The application of this method for varying fiber stiffness was hampered by an inadequate method of measuring fiber stiffness. Initially, fiber stiffness was assumed to be related to the zero-span tensile test. The fibers were assumed to be stiffer if their tensile strength was higher. However, tests of the handsheets made with annealed fibers did not show a variation in zero-span tensile strength. Thus, a decision was made not to rely on the assumption that the zero-span tensile strength is an indirect measure of fiber stiffness. Other methods for determining the fiber stiffness should be developed.

This project did generate information that supports the hypothesis that fiber to fiber bonding has a strong influence on the ultimate compressive strength of the sheet. The relationship between the compressive strength of the sheet and ZDT strength can be represented by a smooth curve for the softwood pulp fibers used in this study and the two methods of pulping the fibers (Fig. 4). The data consist of the ultimate compressive strength of handsheets made from loblolly pine pulped by the kraft process and sheets made from northern jack pine pulped by the chlorite process.

Because the initial attempts to develop the four sets of handsheets were not completely successful, the project was reviewed and a stronger plan has been developed. The two most important features of this revised research plan are the segregation of the fibers into the summerwood and springwood fibers and the development of a direct measurement of fiber stiffness. It is held that summerwood fibers are about three times stiffer than the springwood fibers; therefore, fiber stiffness can be varied by using all summerwood or all springwood fibers.

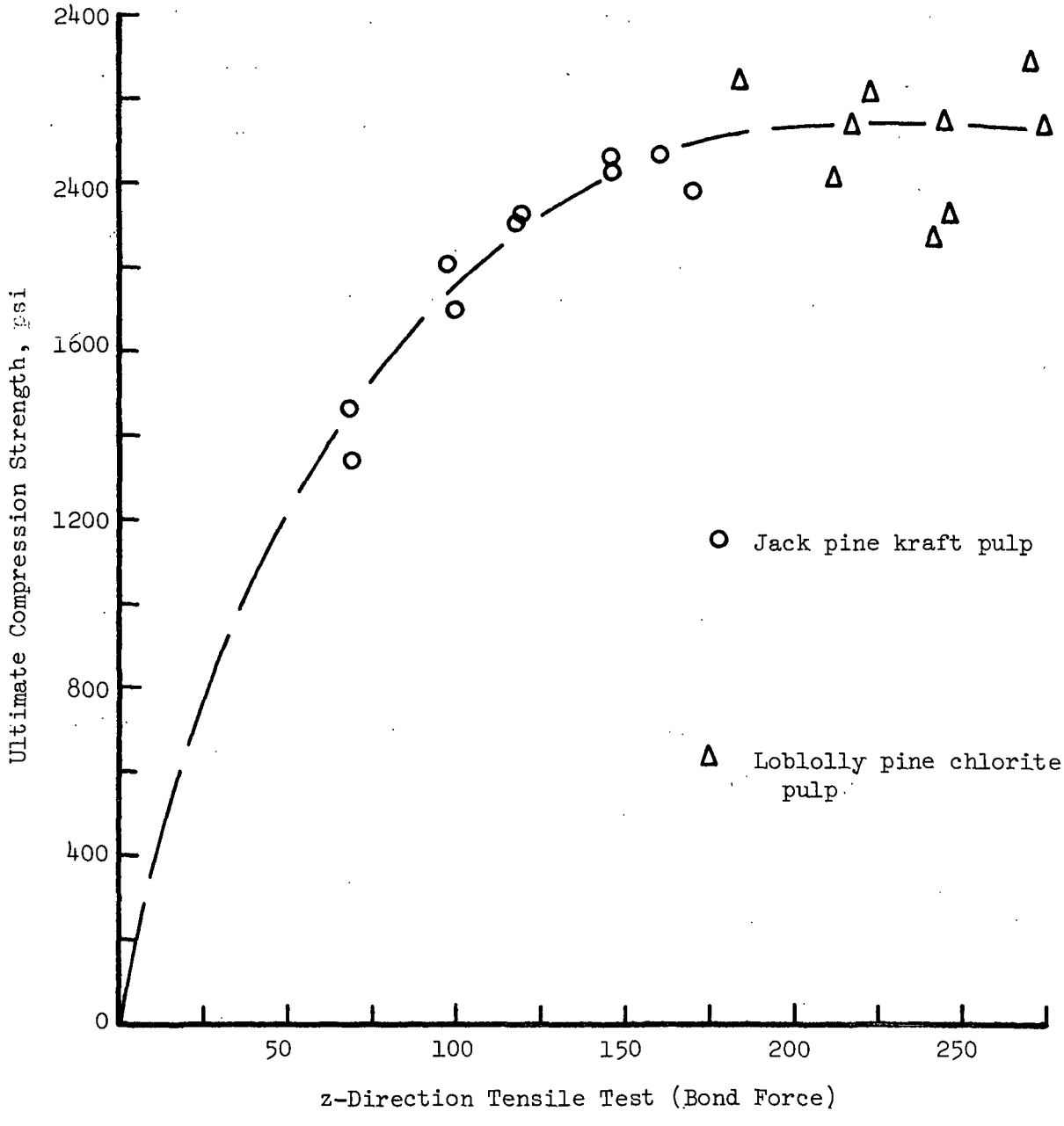

Figure 4. Relationship Between Ultimate Compressive Strength and Bond Force

INTRODUCTION

The importance of the compressive strength of linerboard in the end use performance of corrugated containers has been demonstrated in recent work at the Institute (1). Unfortunately, very little is known about compression failure of linerboard. There is still no universally acceptable method to measure the ultimate compressive strength of linerboard; most of the research to date deals with tensile failure. Although tension is important in sheet formation and manufacturing, once the paper sheets are in the unique structural form of corrugated board, the compressive strength is a key quantity in determining service performance.

Two of the more important articles on tensile strength are those of Van den Akker and Page. Van den Akker (2) indicated that the tensile moduli of a sheet would be expected to depend on both the elastic moduli of the fibers and the distribution and strength of the bonds. Page (3) equated the tensile strength of the sheet to the tensile strength of the fibers and the bond strength between the fibers. It has been the hypothesis that under compression the controlling fiber property would not be the fiber tensile strength but the fiber's resistance to buckling (fiber stiffness). Therefore the role of fiber bonding and fiber buckling in the compressive failure of linerboard needs to be defined. Such knowledge would be helpful in providing a better understanding of what triggers failure and the specific characteristics of the components that need to be modified to improve the compressive strength of corrugated board and boxes.

An outline of the experimental research study that conceptually embraced the preparation of sheets of widely different characteristics in terms of the

degree of bonding and inherent fiber stiffness was prepared (4). Inasmuch as bonding strength is dependent on the number of bonds and the strength of the bonds, equivalent bonding strength may be achieved by any number of arrangements of fiber bonds and bond strength. Accordingly, bond strength is to be expressed as bond density — bonding force per unit relative bonded area.

A central objective for this research has been to produce four sets of handsheets which are to be loaded in compression to determine the role of fiber stiffness and bond strength in resisting compression failure. The handsheets were to be constructed such that the first set had a high bond density and a high fiber stiffness. The second set was to have a high fiber stiffness and a low bond density. The third set would have a low fiber stiffness and a high bond density, and the fourth set would have a low bond density and a low fiber stiffness.

$$\left[\text{High Fiber Stiffness}\right]_1 \left(\frac{\text{High Bond Force}}{\text{Bond Area}}\right)_1$$

Handsheet Set 1

$$\left[\text{High Fiber Stiffness}\right]_1 \left(\frac{\text{Low Bond Force}}{\text{Bond Area}}\right)_2$$

Handsheet Set 2

$$\left[\text{Low Fiber Stiffness}\right]_2 \left(\frac{\text{High Bond Force}}{\text{Bond Area}}\right)_1$$

Handsheet Set 3

$$\left[\text{Low Fiber Stiffness}\right]_2 \left(\frac{\text{Low Bond Force}}{\text{Bond Area}}\right)_2$$

Handsheet Set 4

The physical properties of these handsheets were to be measured. It is postulated that handsheets prepared with these properties would fail in the

following manner. A sheet having a high bond density and a low fiber stiffness would be expected to have the compression failure initiated by buckling of the individual fibers. A sheet that has a high fiber stiffness and a low bond density would be expected to have the compression failure initiated by bond breakage and a delamination of the sheet. Handsheets that have a high fiber stiffness and a high bond density or sheets with a low fiber stiffness and a low bond density would have the compression failure initiated by a combination of bond breakage and fiber buckling.

The full set of evidence generated by this project to determine the role in compression of fiber stiffness and fiber to fiber bonding was to be: the four sets of handsheets with their unique combination of fiber stiffness and bond strength, the ultimate compressive strength of the sheets, photomicrographs of the sheets that have failed in compression, and high speed photomicrographs of the edge of the liner as the ultimate compressive load is reached.

HANDSHEET DEVELOPMENT

Current knowledge is not sufficient to allow manipulation of the basic methods of sheet formation to produce a set of handsheets with essentially the same fiber stiffness and varying bond strength, nor to produce a set of handsheets with essentially the same bond density and various fiber stiffnesses. Present knowledge does not include how far the bond strength and fiber stiffness must be changed to produce a handsheet wherein the fibers buckle before bond failure occurs or, conversely, to produce handsheets where the bonds fail before the fibers buckle. Consequently, the first consideration in carrying out the research program was the determination of the feasibility of making handsheets which vary the bond density at constant fiber stiffness and vary the fiber stiffness at constant bond density.

VARYING BOND DENSITY

Much is known about bonding of papermaking fibers; however, very little work has been carried out on ways of changing fiber stiffness. Consequently, care must be exercised that the methods of varying interfiber bond density do not significantly change fiber stiffness. For the purpose of determining feasible ways of making handsheets of varying bond density at constant fiber stiffness, a sample of unbleached southern pine kraft pulp was refined at 1.5% consistency in a laboratory Valley beater to approximately 350 ml Canadian Standard freeness (C.S.F.). Two such beater runs were made to provide the desired quantity of pulp. The total pulp refined above was classified in a Bauer-McNett fractionation apparatus using 10-, 35- and 65-mesh screens. The on-35-mesh stock collected in the above fractionation was refractionated and the stock retained on the on-10-mesh screen was saved. The on-10 fractions obtained above were combined and refractionated

using the 10-, 35- and 65-mesh screens. The on-35 fraction was found to give a yield of 21% and was saved for use in subsequent trials in which wet pressing was used to vary bond density. It is well known that bond density increases with increasing wet pressing. Within normal range, wet pressing would not be expected to affect the fiber stiffness.

Using the twice classified on-35-mesh stock, two sets of handsheets were made on an 8 x 8 inch handsheet mold. The first set of sheets was wet pressed at three pressure levels, namely 10, 50 and 100 psi for 5 minutes. The second set of handsheets was formed from stock containing 2% Cato 15 starch. The starch was blended for 5 minutes before the sheets were formed. The sheets containing the 2% starch were wet pressed at 100 psi for 5 minutes. In both sets the desired level of wet pressing was obtained as quickly as possible (referred to as instantaneously) in contrast to the gradual application of the pressure. The latter was used in subsequent trials as a means of avoiding any disrupture of the web due to the too rapid movement of the water in the web. The handsheets formed were conditioned and evaluated for physical properties including bond density.

Since the range of bond density obtained using 10, 50 and 100 psi wet pressing was considered inadequate, additional trials were made using higher and lower levels of wet pressing and in two cases a debonding agent (0.1% Hyamine). Inasmuch as the objective of this phase of the study was to determine the feasibility of making handsheets of varying bond density at constant fiber stiffness, it was decided that feasibility could be established as readily on an unfractionated run-of-the-mill unbleached linerboard pulp. This would result in a considerable saving in time and, more importantly, a saving in the specific southern pulp obtained for use in the experimental research phase of the study. Accordingly,

the additional trials, using higher and lower levels of wet pressing and a debonding agent, were made using a run-of-the-mill unbleached northern jack pine kraft pulp, refined in a Valley beater to 350 ml C.S.F. The level of wet pressing and debonding agent used in these latter trials is shown in Table I.

TABLE I

NORTHERN JACK PINE HANDSHEETS — SAMPLE NUMBERS AND WET PRESSING PRESSURE (3 PLY)

Sample No.	Wet Pressing, psi	Hyamine, %
1-H	1	0.1
10-H	10	0.1
10-i	10	0
10-g	10	0
50-i	50	0
50-g	50	0
100-i	100	0
100-g	100	0
250-i	250	0
250-g	250	0
500-i	500	0
500-g	500	0

The handsheets were prepared by the following method. The on-35 fraction of the fractionated pulp was diluted to 1.5% consistency and dispersed in a British disintegrator for 1500 revolutions after which it was diluted to 0.25% consistency. Additives, when used, were added at this point. Sufficient stock to yield a 2.625 g

oven dried sheet was measured and formed into a handsheet on a Noble and Wood sheet machine. The handsheet was vacuum couched onto a premoistened blotter. A second and third ply were formed in the same manner and vacuum couched onto the previous couched sheet. The 3-ply sheet was then covered with a premoistened blotter for subsequent wet pressing. Single-ply sheets were formed in the same way for zero-span tensile testing. Deionized water was used throughout the sheet-making process. The handsheets were drum dried and then conditioned before testing. The handsheets made of the unbleached northern jack pine kraft were prepared in the same way as described above.

Handsheet Evaluation

All handsheets were conditioned and tested at standard conditions (50% RH, 23°C) with the exception of the optical tests which were carried out at 45% RH, 24°C.

The physical tests are described below:

1. Basis weight; four different areas of the sheets were weighed for determination of basis weight:

   a. The entire 8 x 8 inch sheet was weighed on a basis weight scale,
   b. The circular z-direction tensile specimens, having an area of 6.682 sq cm, were weighed on an analytical balance,
   c. The edgewise compression specimens, having an area of 54.02 sq cm, were weighed on an analytical balance, and

d. Circular specimens cut adjacent to the edgewise compression failure line and having an area of 2.896 sq cm were weighed on an analytical balance.

The basis weight determined in (d) above has the best chance of approximating the basis weight at the center of the edgewise compression specimen.

2. Caliper. The caliper was measured with a micrometer meeting the requirements of TAPPI Method T 411 os-76. Two measurements of caliper were made for each sheet — one at the center of the edgewise compression specimen and one at the center of the z-direction tensile specimen.

3. z-Direction tensile strength. The z-direction tensile test measures the interfiber bond strength of paper. The z-direction tensile strength was by a procedure developed by Wink and Van Eperen (5). In this method the specimen is adhered, by means of an epoxy adhesive, to the ground surface of metal cylinders having an area of 4.969 sq cm. The adhesive is allowed to cure for 16 or more hours, and the force necessary to separate the cylinders is measured with a tensile testing machine.

4. Zero-span tensile strength. The zero-span tensile strength test is used to directly measure fiber strength in terms of the force to rupture. It does not measure flexural stiffness; however, an initial assumption was made that the greater the zero-span tensile strength, the greater the flexural stiffness. Zero-span tensile strength was measured with an IPC zero-span instrument described by Wink and Van Eperen (6). The measurement was made on single-ply handsheets having a basis weight of approximately 68 g/m^2. The basis weight of a circular specimen, including the zero-span test zone, having an area of 2.896 cm^2 was measured and used to compute the zero-span breaking length.

5. Compression strength of the handsheet. The edgewise compression strength was measured with a compression instrument developed by Weyerhaeuser Co. In this instrument the specimen is firmly clamped at each end and laterally supported on each side by closely spaced stabilizing blades. The blades are mounted as cantilever beams with the free end adjacent to the test specimen. Spacing between adjacent blades was 0.130 inch resulting in a slenderness ratio $(1/(I/A))^{1/2}$ of 22, 20 and 32 for the specimen thickness of the sheets pressed at 10, 50 and 100 psi. The specimen was necked down in the central area to give a minimum width of 0.79 inch. The test specimens were loaded at a rate of 0.08 inch per minute. The compression strength is given as the maximum load sustained before failure divided by the minimum cross-sectional area.

6. Bonding density — bonding force per unit bonded area. The strength of paper is primarily a function of (a) the intrinsic strength of the individual fiber, (b) the number of interfiber bonds and (c) the strength of the interfiber bonds. It may be recalled that bond strength as used in this study is to be expressed as bond density — bond force per unit relative bonded area. The latter may be obtained by dividing the interfiber bond force by the relative bonded area. The determination of interfiber bonding force is described in (3) above. The method used in this study for determining relative bonded area is as follows:

In a given sheet, the fibers have a total surface area available for potential bonding. All the available fiber area, however, is not in bonding contact; consequently, only that portion of the total fiber area which is in bonding contact represents the total bonded area. The portion which is not in bonding contact is referred to as total unbonded area. The above relationship

may be represented by

$$A_t = A_b + A_u \qquad (1)$$

where

A_t is the total fiber area

A_b is the bonded fiber area

A_u is the unbonded fiber area

One of the most widely used methods employed for estimating the amount of bonded area in paper is the so-called optical technique originally studied by Parson (7) and later explored by Ratliff (8), who compared optical measurements with corresponding measurements made by means of a gas absorptive method. The gas absorptive method is generally considered to be more accurate; however, it is more time consuming. The optical method was used because it is considered to give a reasonable estimate of bonded area especially when comparative data on the same furnish are involved.

The optical method involves numerical values of the specific scattering coefficient which may be calculated from the original theory of Kubelka and Munk (9,10). The Kubelka and Munk theory quantitatively relates the scattering and absorption of light to the diffuse reflectance of light-scattering material such as paper. The most convenient form of the theory as adapted to paper may be found in equations and charts presented by Steel (11) and Judd (12). In the original work by Judd, the specific scattering coefficient and specific adsorption coefficient are related to thickness. Van den Akker (13) has redefined these two parameters in terms of basis weight of paper (in c.g.s. units) which are more useful. The relationships developed by Van den Akker quantitatively relate the scattering and absorption of light to the transmittance and reflectance of a light scattering paper in terms of basis weight of the paper.

The procedure involves measuring any two of the three optical properties, $\underline{R}_o$, the reflectance of sheet backed by a black body, $\underline{R}_\alpha$, the reflectance of a sheet backed by a thick pad of the same material, and $\underline{T}$, transmittance of a single sheet of the paper. Unbleached linerboard is virtually opaque in the visible range of monochromatic light using instruments such as the General Electric Recording Spectrophotometer or the Brightness Tester. Therefore, these instruments could not be used satisfactorily for measuring the optical properties. Unbleached linerboard, however, is less opaque to wavelengths above the visible range; thus the two independent optical properties $\underline{R}_o$ and $\underline{T}$ were measured on a Beckman DK-2A infrared recording spectrophotometer. A typical pair of reflectance ($\underline{R}_o$) and transmittance ($\underline{T}$) curves for 42-lb sheets is shown in Fig. 5. The desired optical measurements were read at 1150 nm wavelength. The $\underline{R}_o$ and $\underline{T}$ together with basis weight were used in a computer program to calculate the specific scattering coefficient in cm^2/g.

The relative bonded area may be computed from light scattering measurements based on the following relationships:

- The specific scattering coefficient, $\underline{S}_u$, of a sheet dried from water varies linearly with the unbonded fiber surface area, $\underline{A}_u$, according to the following equation:

$$S_u = k\, A_u + i \qquad (2)$$

where

$\underline{S}_u$ is the specific scattering coefficient of the sheet of paper

$\underline{A}_u$ is the unbonded area, and

$\underline{k}$ and $\underline{i}$ are constants

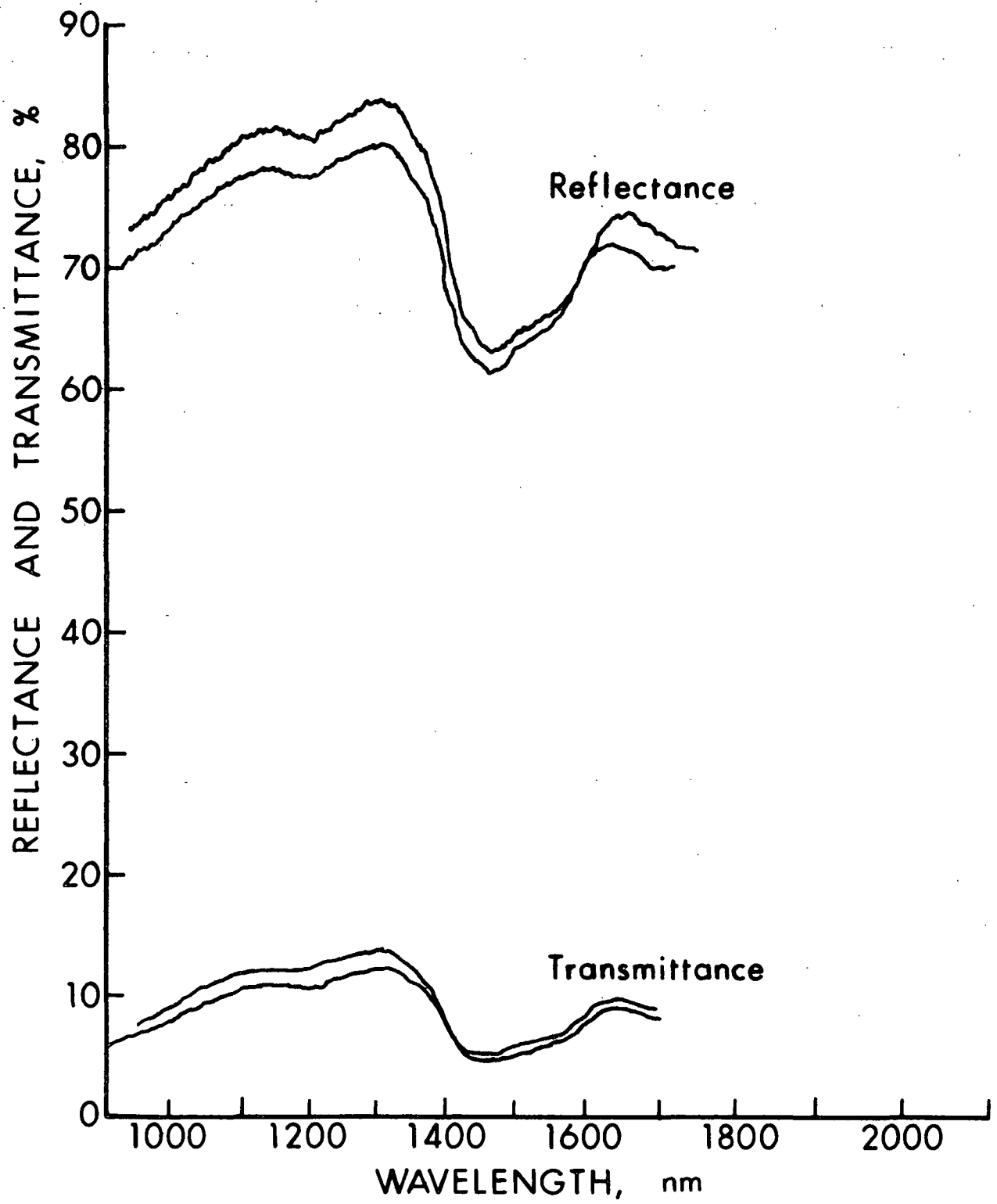

Figure 5. Typical Reflectance and Transmittance Curves

- The specific scattering coefficient of totally unbonded fibers $\underline{S_t}$ bears a similar relationship to the total possible exposed fiber surface area $\underline{A_t}$,

$$S_t = k A_t + i \tag{3}$$

- Thode and Ingmanson (14) found that the total dry fiber surface area available for fiber bonding which is effective in developing physical strength, e.g., tensile strength, remains constant with refining and is unaffected by production of fines or the degree of fibrillation. This permits extrapolation of a plot of $\underline{S_u}$ versus tensile (or compression strength) to zero tensile strength to obtain an estimate of specific scattering coefficient of totally unbonded fiber, $\underline{S_t}$.

Since the bonded area, $\underline{A_b}$, is by definition the difference between the total possible surface area $\underline{A_t}$ and the unbonded surface area, $\underline{A_u}$:

$$A_b = A_t - A_u = (S_t - S_u)/k \tag{4}$$

or

$$k A_b = k (A_t - A_u) = S_t - S_u \tag{5}$$

where the quantity $\underline{k}\ \underline{A_b}$ is proportional to the bonded area.

The relative bonded area then becomes

$$\frac{A_b}{A_t} = \left(\frac{S_t - S_u}{S_t} \right) \tag{6}$$

where

$\underline{A_b}$ is the fiber bonded area

$\underline{A_t}$ is the total possible fiber surface area

$\underline{S_t}$ is the specific scattering coefficient of totally unbonded fibers

$\underline{S_u}$ is the specific scattering coefficient of the sheet of paper

Once the optical properties of reflectance and transmittance are available, the scattering coefficient of the totally unbonded fibers, $\underline{S_t}$, can be obtained by extrapolation of a plot of $\underline{S_u}$ versus compression strength to zero compression strength to obtain $\underline{S_t}$, the scattering coefficient of a completely unbonded sheet. Figure 6 shows the extrapolated technique. Bond density was obtained by dividing the relative bonded area by the bond force determined by means of the z-direction tensile test.

Discussion of Results

It may be recalled that the objective in this phase of the research was to determine the feasibility of making kraft handsheets at varying levels of bond density at constant fiber stiffness. The bonding is to be expressed as bond density — bond strength per unit relative bonded area. The fiber property of concern is flexural stiffness and is measured indirectly in terms of the zero-span tensile test.

A great deal is known about variables which affect fiber bonding; however, little is known as to how to change fiber stiffness or the effect of bonding agents on fiber stiffness. A number of approaches to varying the bonding over a reasonable range at constant fiber stiffness were considered. However, the most appealing approach in view of the constant fiber stiffness requirement has been

to use wet pressing as a means of varying the bond density with little effect, if any, on fiber stiffness.

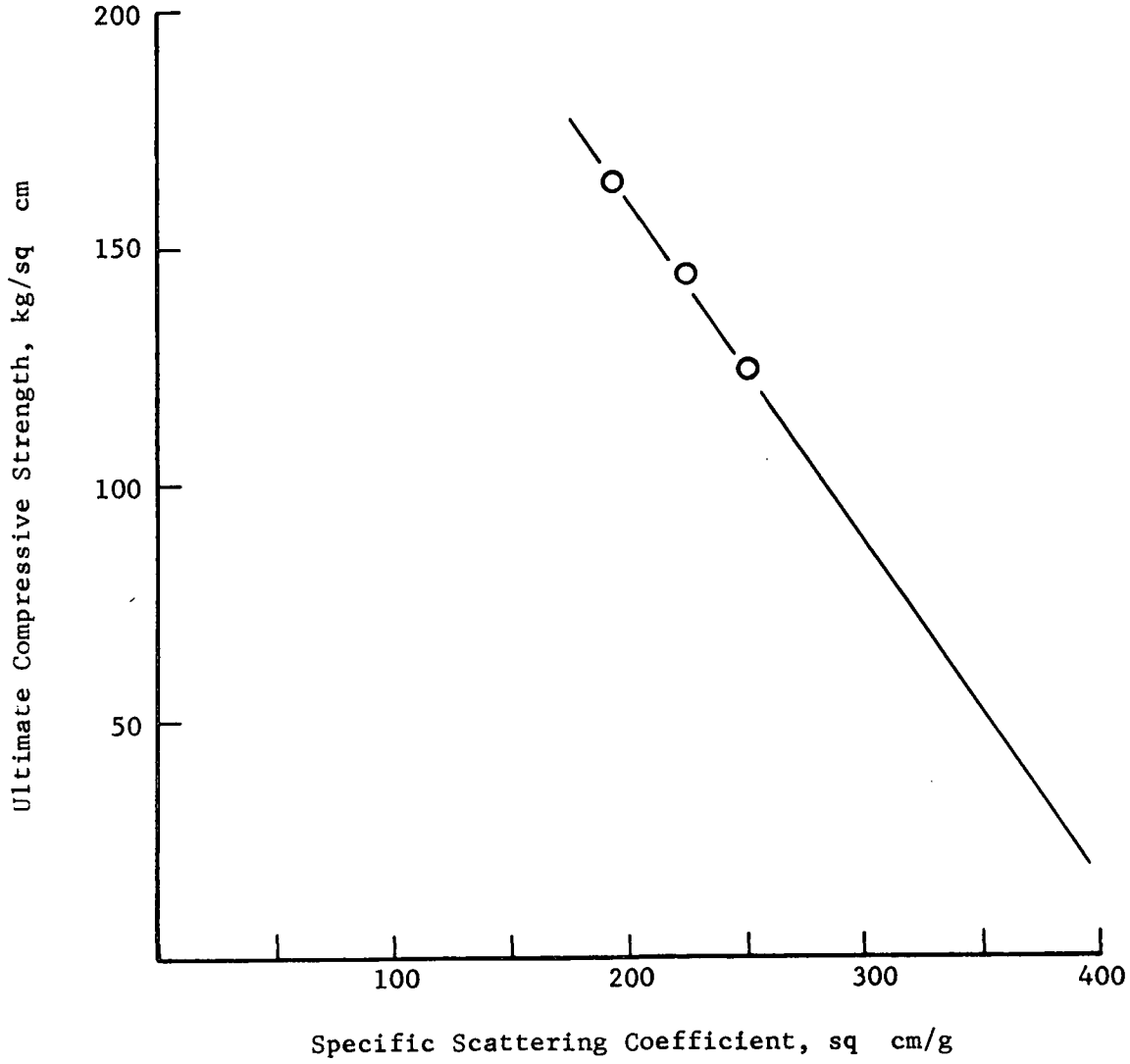

Figure 6. Extrapolation Technique Obtaining Scattering Coefficient of Totally Unbonded Sheet

In the first series of trials in this area, the fractionated unbleached southern pine linerboard pulp was refined to 350 ml C.S.F. and fractionated on a Bauer-McNett fractionator. The through-10 on-35 fraction was subjected to a second fractionation and the fibers retained on the 35-mesh screen made into 3-ply wet laminated 42-lb handsheets. The handsheet results are given in Table II. It

TABLE II

PHYSICAL PROPERTIES OF HANDSHEETS
(Southern Pine Unbleached Kraft Pulp)

Sample Identification Number	Wet Pressing (psi)	Ultimate Compression (psi)	Basis Weight (lb/M ft^2)	Caliper (mil)	Scattering Coefficient (cm^2/g)		Relative Bonded Area (curve fit) (%)	Fiber Strength Zero-Span Tensile (km)	Relative Bond Strength ZDT (ZDT/RBA) (psi)
					Before Compression	After Compression			
10-A	10	1758	43.3	19.9	250.4	250.2	29.5	14.32	24.1 (81.7)
50-A	50	2055	43.4	16.4	224.2	225.6	36.9	14.42	41.7 (113.0)
100-A	100	2331	43.1	14.0	193.5	196.5	45.5	15.25	49.3 (108.4)
1005[b]-A	100	2198	43.3	14.5	195.4	199.4	45.0	14.6	60.8 (135.1)

[a]All physical properties corrected for basis weight except caliper.

[b]Contained 2% Cato starch 15.

may be noted that the relative bonded area increased from 0.295 to 0.455 when the wet pressing was increased from 10 to 100 psi. Similarly, the z-direction tensile which is a measure of bonding force increased from 24.1 to 49.3. When these results are used to calculate bond density — bond force per unit relative bonded area — the bond density of the handsheets wet pressed at 100 psi was lower than at 50 psi wet pressing. It would be expected that the bond density should increase progressively with the level of wet pressing until web damage occurs. Increasing wet pressing increases bonding by increasing the possibility that more fiber segments or areas of fiber segments are brought into the field of molecular attraction or bonding. The scattering coefficient decreases with increases in wet pressing, as would be expected because the scattering will decrease as fiber-to-fiber contact increases. Ultimate compression strength increased with an increase in the level of wet pressing. It may be noted that the fiber stiffness measured in terms of zero-span tensile did not change significantly with change in the level of wet pressing.

Photomicrographs of the compression failure zone for the samples with a wet pressing pressure of 10 psi and 100 psi both show a failure zone which appears to be caused by bond failure (Fig. 7 and 8).

The addition of 2% Cato starch increased the bond density significantly as would be expected; however, the ultimate compression strength was lower than the corresponding handsheets made without the starch, thus the compression value for the handsheets with 2% starch are suspect. The results show no marked change in fiber strength.

The compression strength was plotted against the specific scattering coefficient, and the curve was extrapolated to zero compression strength to

obtain an estimate of the scattering coefficient of a totally unbonded sheet. The plot is shown in Fig. 9. The intercept on the abscissa is approximately 410 sq cm/g; however, the points are too close together to give a good estimate of the intercept.

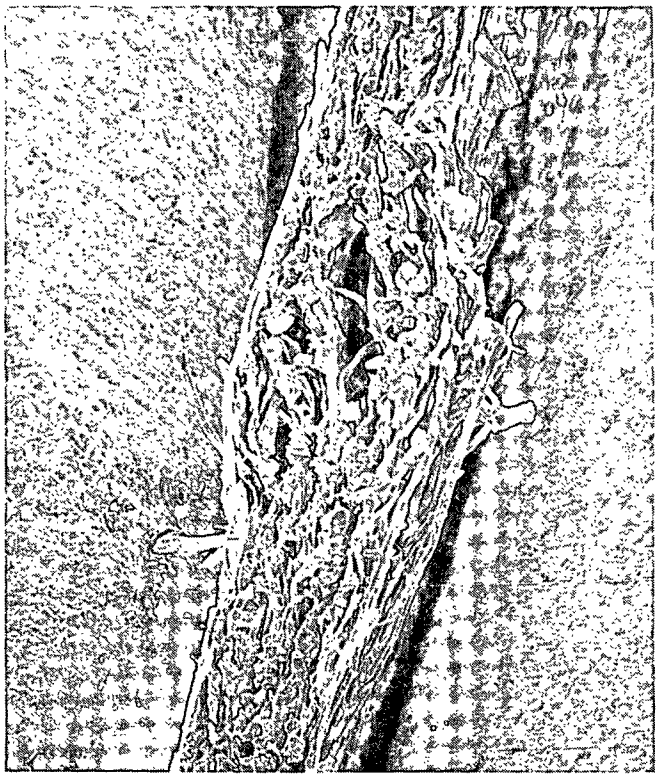

Figure 7. Compressive Failure of Handsheets Formed with 10 psi Wet Pressing

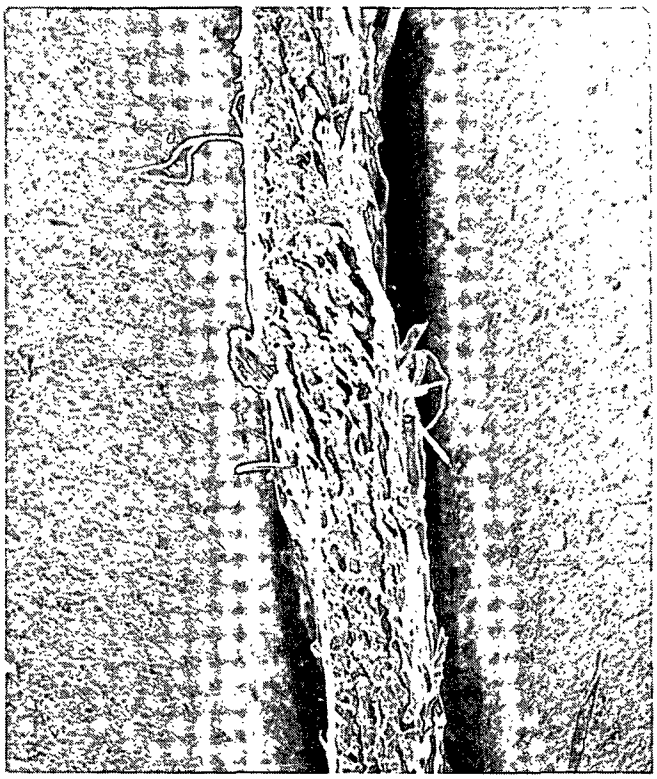

Figure 8. Compressive Failure of Handsheets Formed with 100 psi Wet Pressing

In order to expand the range of bond density and to improve the reliability of the extrapolation of the scattering coefficient of a totally unbonded sheet, additional trials were carried out. Because of the low yield (21%) of the on-35-mesh pulp resulting from the double fractionation, it was decided to carry out further exploratory trials using an unbleached run-of-the-mill kraft pulp. For this purpose an unbleached northern jack pine kraft pulp was selected, refined, and made into three-ply wet laminated handsheets. The handsheets were wet pressed

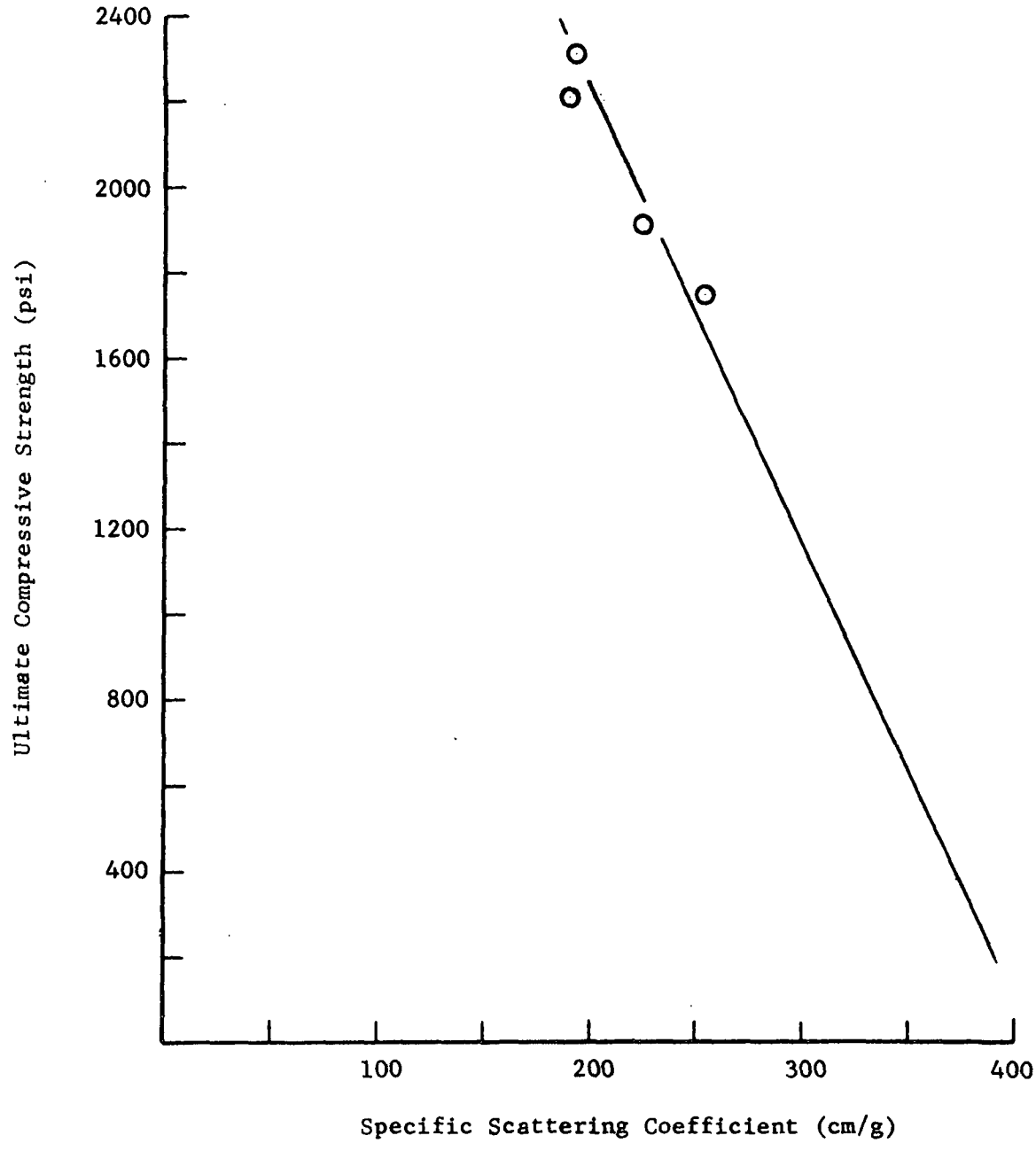

Figure 9. Relationship Between Compression Strength and Specific Scattering Coefficient (Southern Pine Unbleached Kraft)

at 10, 50, 100, 250 and 500 psi. The pressure was applied in two ways. In one case the pressure was applied instantaneously and held at the desired level for 5 minutes. In the other case, the pressure was applied gradually so as to reduce the initial rate of water removal. In the latter case, five minutes were used to reach the desired pressure level, and the maximum pressure was applied for 5 additional minutes. The handsheets made with gradual and instantaneously applied wet pressing are identified with the suffix g and i, respectively. The results obtained on these handsheets are tabulated in Table III. Examination of the zero-span tensile data indicates that there was no significant change in the fiber strength with change in the magnitude of the wet pressing. The effect of the gradual versus instantaneous application of the wet pressing pressure had no significant effect on the results. It was anticipated that the gradual application might be the better way in that the pressure would be applied for a longer time and the impact of the water being expelled from the wet web would be less.

The compression strength increased with the level of wet pressing up through 250 psi wet pressing (Fig. 10). There was very little difference between 250 and 500 psi wet pressing. As would be expected, the scattering coefficient decreased with an increase in wet pressing and hence, bonding. The realtive bonded area also showed the expected trend of increasing with increase in wet pressing.

The relative bond force determined by the z-direction tensile test increased with wet pressing (Fig. 11). This would be expected. The bond density which, it may be recalled, is the bond force per unit of relative bonded area also increased with the degree of wet pressing. Varying the wet pressing from 10 to 500 psi resulted in a range of bond density from approximately

TABLE III

PHYSICAL PROPERTIES OF HANDSHEETS
(Northern Jack Pine Unbleached Kraft Pulp)

Sample Identification Number	Wet Pressing (psi)	Ultimate Compression (kg/cm^2) (psi)	Basis Weight ($lb/M\ ft^2$)	Caliper (mil)	Scattering Coefficient (cm^2/g) Before Compression	Scattering Coefficient (cm^2/g) After Compression	Relative Bonded Area (linear fit) (%)	Fiber Strength Zero-Span Tensile (km)	Relative Bond Strength ZDT (ZDT/RBA) (psi)
10-i	10	(102.72) 1461	44.0	13.9	258.76	251.52	38.4	16.82	67.6 (176.0)
10-g	10	(92.02) 1323	44.1	14.2	266.34	249.7	36.6	---	63.7 (174.0)
50-i	50	(120.09) 1708	43.5	11.6	221.62	226.74	47.2	17.53	97.4 (206.4)
50-g	50	(128.66) 1830	44.6	11.9	227.52	224.09	45.8	---	94.5 (206.3)
100-i	100	(134.36) 1911	43.3	10.9	206.41	204.20	50.8	17.29	116.0 (228.3)
100-g	100	(135.48) 1927	43.2	10.8	204.0	205.21	51.4	---	117.0 (227.6)
250-i	250	(145.68) 2072	42.7	10.1	180.62	181.36	57.0	---	145.0 (254.4)
250-g	250	(143.64) 2043	43.5	10.2	177.62	152.35	57.7	---	145.0 (251.3)
500-i	500	(145.82) 2074	42.6	9.8	168.55	160.84	59.9	18.07	158.0 (263.8)
500-g	500	(139.49) 1984	41.5	9.8	164.6	164.36	60.8	---	171.0 (281.3)

[a]All physical properties corrected for basis weight except caliper.

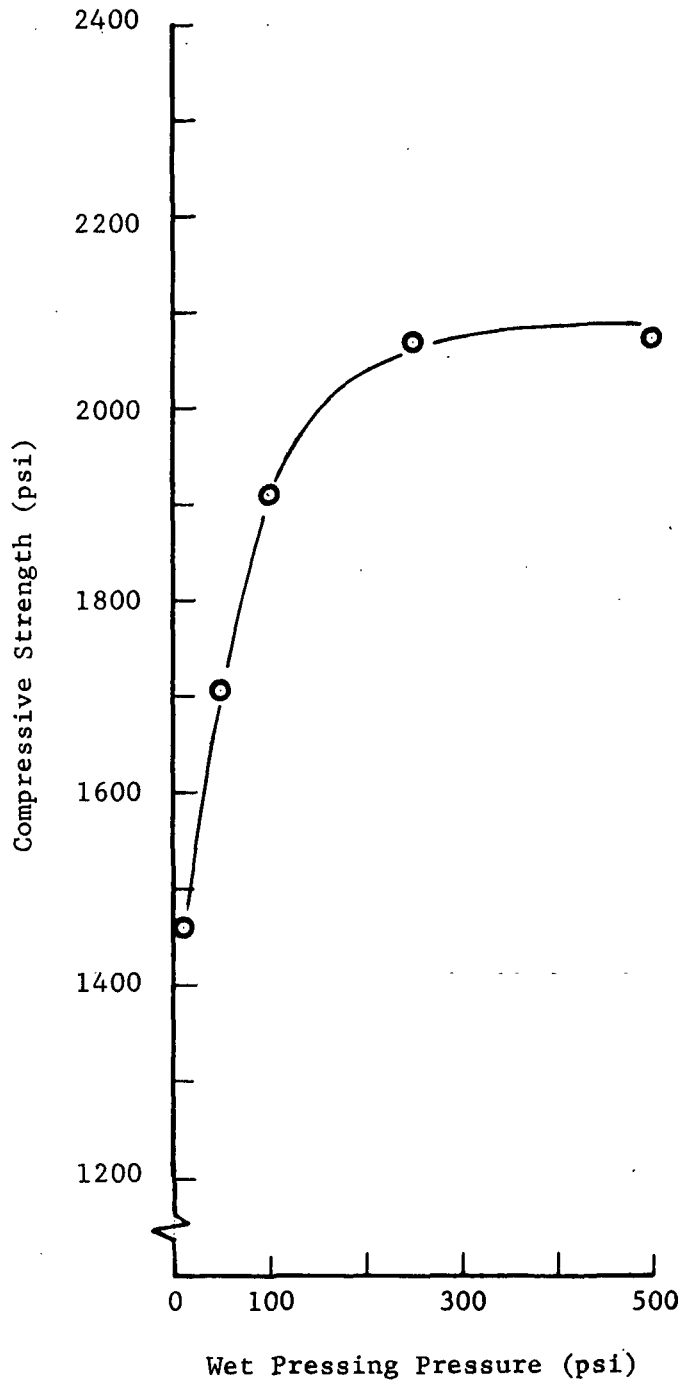

Figure 10. Relationship Between Sheet Ultimate Compressive Strength and Wet Pressing Pressure (Northern Jack Pine Unbleached Kraft Pulp)

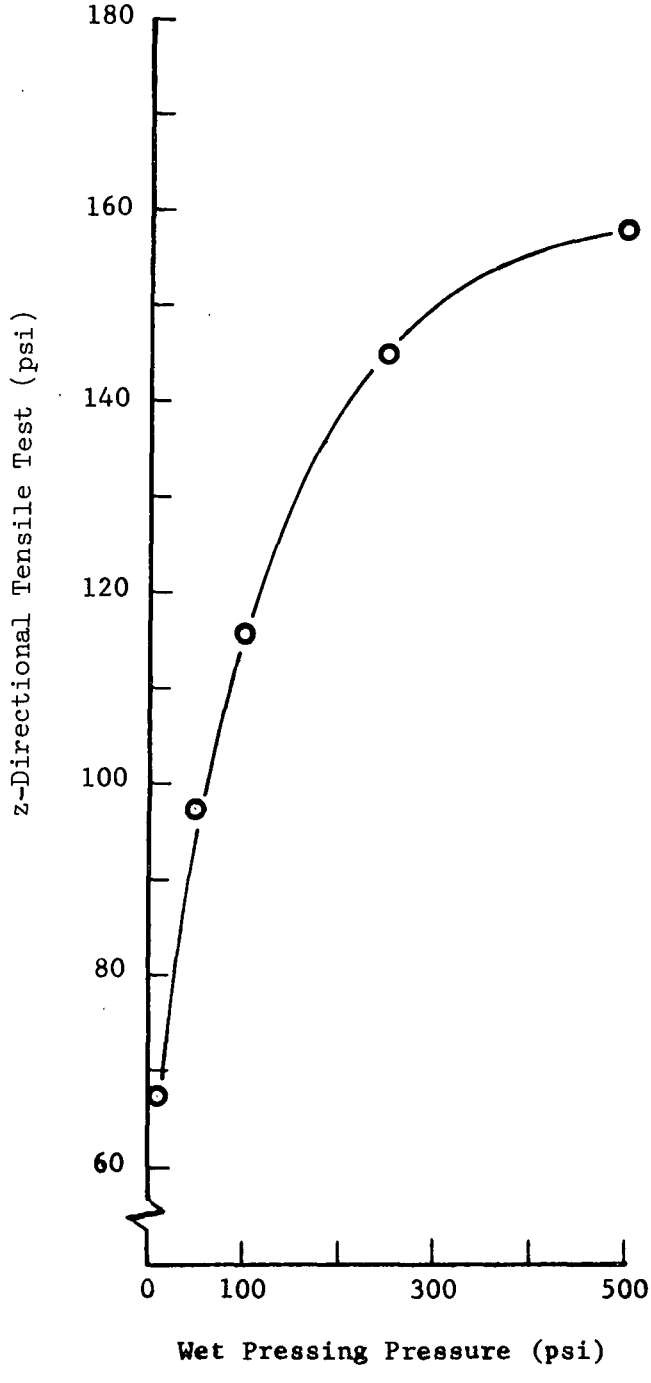

Figure 11. Relationship Between z-Directional Tensile Strength and Wet Pressing Pressure (Northern Jack Pine Unbleached Kraft Pulp)

175.0 to 273.0 psi (Fig. 12). This range may well be adequate for determining the effect of varying bonding at constant fiber stiffness on compression failure.

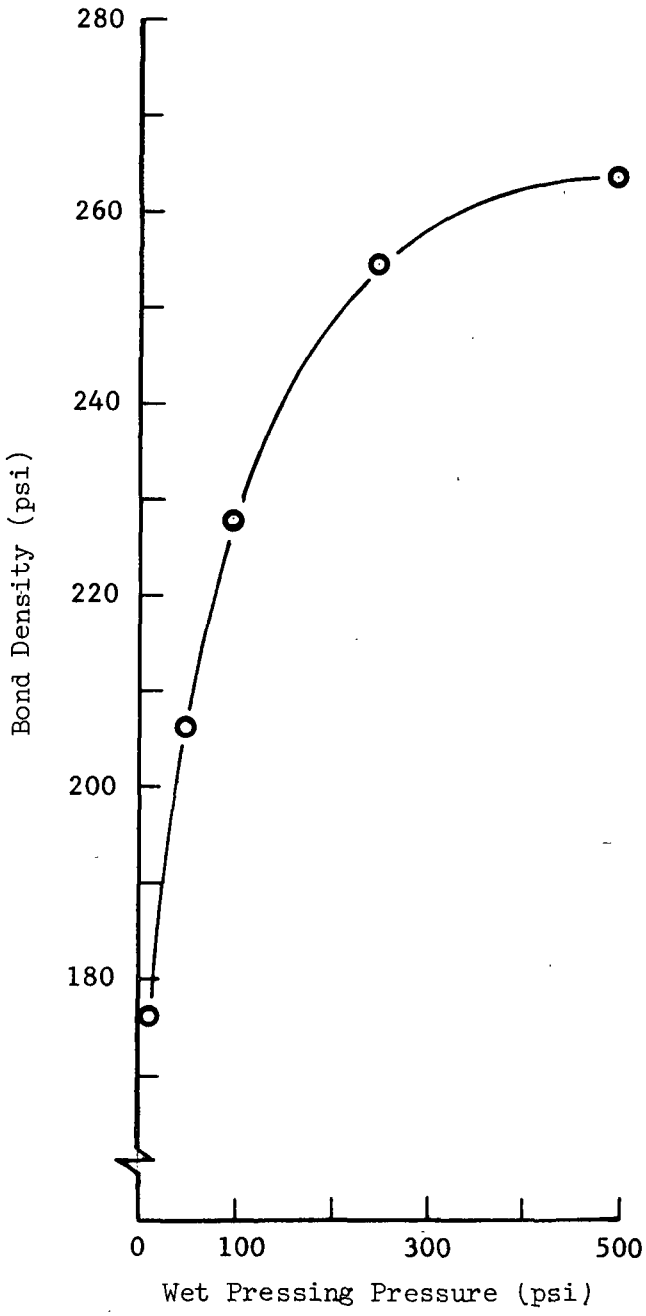

Figure 12. Relationship Between Bond Density and Wet Pressing Pressure (Northern Jack Pine Unbleached Kraft Pulp)

As in the first trial, the compression strength was plotted against the specific coefficient, and the curve was extrapolated to zero compression strength to obtain an estimate of the scattering coefficient of the totally unbonded sheet (Fig. 13). The intercept on the abscissa is approximately 420 sq cm/g. The points defining the line of intercept are clustered about a rather small area, and thus any small deviation in drawing the line through these points can have a marked effect on the intercept. Because of this, two additional trials were made using lower wet pressing pressure and/or a debonding agent (0.1% Hyamine). It was hoped that these two trials would provide points on the curve nearer the intercept. The results are tabulated in Table IV. The use of the lower pressure and the debonding agent did not significantly affect the fiber strength as measured by the zero-span tensile test. In contrast, the ultimate compression strength at 1 psi wet pressing together with 0.1% debonding agent was markedly reduced. The results obtained at 10 psi wet pressing and 0.1% debonding agent are out of line with the corresponding results obtained on handsheets wet pressed at 10 psi and containing no debonding agent. The caliper results also lend support to the possibility that the 10-psi results are in error because the presence of the debonding agent should have resulted in a higher caliper than was obtained. The results obtained for scattering coefficient and relative bonded area support the above hypothesis. The results obtained on the 1-H handsheets appear to be more in line with what would be expected.

A plot of the ultimate compression strength versus the specific scattering coefficient is illustrated in Fig. 14. The additional samples did not improve the "accuracy" of the extrapolation. The plotted points appear to fit a curvilinear line more readily than a linear line.

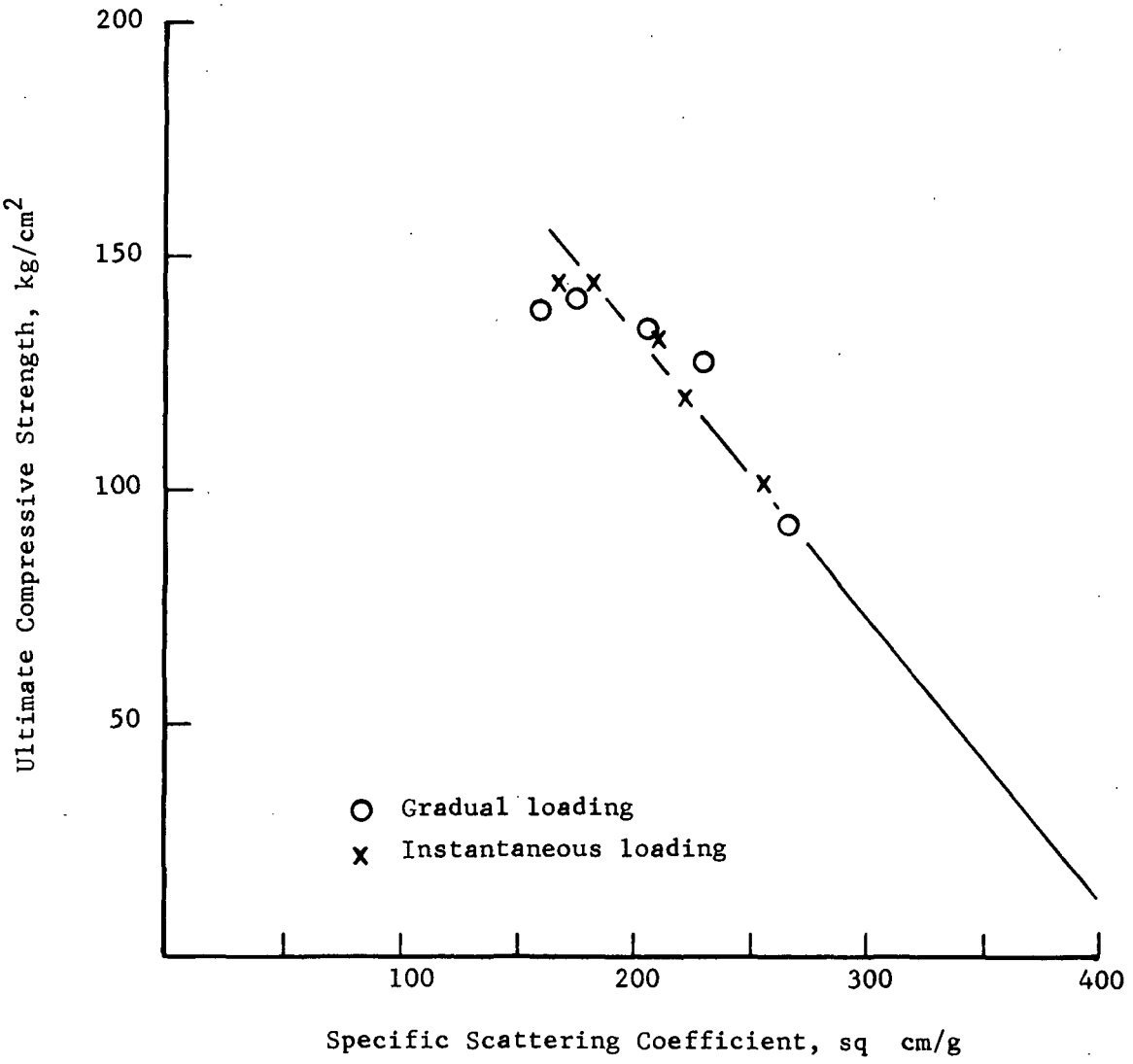

Figure 13. Relationship Between Compression Strength and Specific Scattering Coefficient (Northern Jack Pine Unbleached Kraft Pulp)

TABLE IV

PHYSICAL PROPERTIES OF HANDSHEETS
(Northern Jack Pine Unbleached Kraft Pulp)

Sample Identification Number	Wet Pressing (psi)	Ultimate Compression (kg/cm^2) (psi)	Basis Weight ($lb/M\ ft^2$)	Caliper (mil)	Scattering Coefficient (cm^2/g) Before Compression	Scattering Coefficient (cm^2/g) After Compression	Relative Bonded Area (curve fit) (%)	Fiber Strength Zero-Span Tensile (km)	Relative Bond Strength ZDT (ZDT/RBA) (psi)
10-i	10	(102.72) 1461	44.0	13.9	258.76	251.52	38.4	16.82	67.6 (176.0)
10-g	10	(92.02) 1323	44.1	14.2	266.34	249.71	36.6	---	63.7 (174.0)
50-i	50	(120.09) 1708	43.5	11.6	221.62	226.74	47.2	17.53	97.4 (206.4)
50-g	50	(128.66) 1830	44.6	11.9	227.52	224.09	45.8	---	94.5 (206.3)
100-i	100	(134.36) 1911	43.3	10.9	206.41	204.20	50.8	17.29	116.0 (228.3)
100-g	100	(135.48) 1927	43.2	10.8	204.0	205.21	51.4	---	117.0 (227.6)
250-i	250	(145.68) 2072	42.7	10.1	180.62	181.36	57.0	---	145.0 (254.4)
250-g	250	(143.64) 2043	43.5	10.2	177.62	182.35	57.7	---	145.0 (251.3)
500-i	500	(145.82) 2074	42.6	9.8	168.55	160.84	59.9	18.07	158.0 (263.8)
500-g	500	(139.49) 1984	41.5	9.8	164.6	164.36	60.8	---	171.0 (281.3)
1-H (+ .1% Hyamine)	1	(78.3) 1113	43.9	18.4	---	273.33	34.9	16.99	38.4 (110.0)
10-H (+ .1% Hyamine)	10	(113.0) 1603	44.6	13.8	---	250.15	40.4	16.91	71.2 (176.2)

[a]All physical properties except caliper corrected for basis weight.

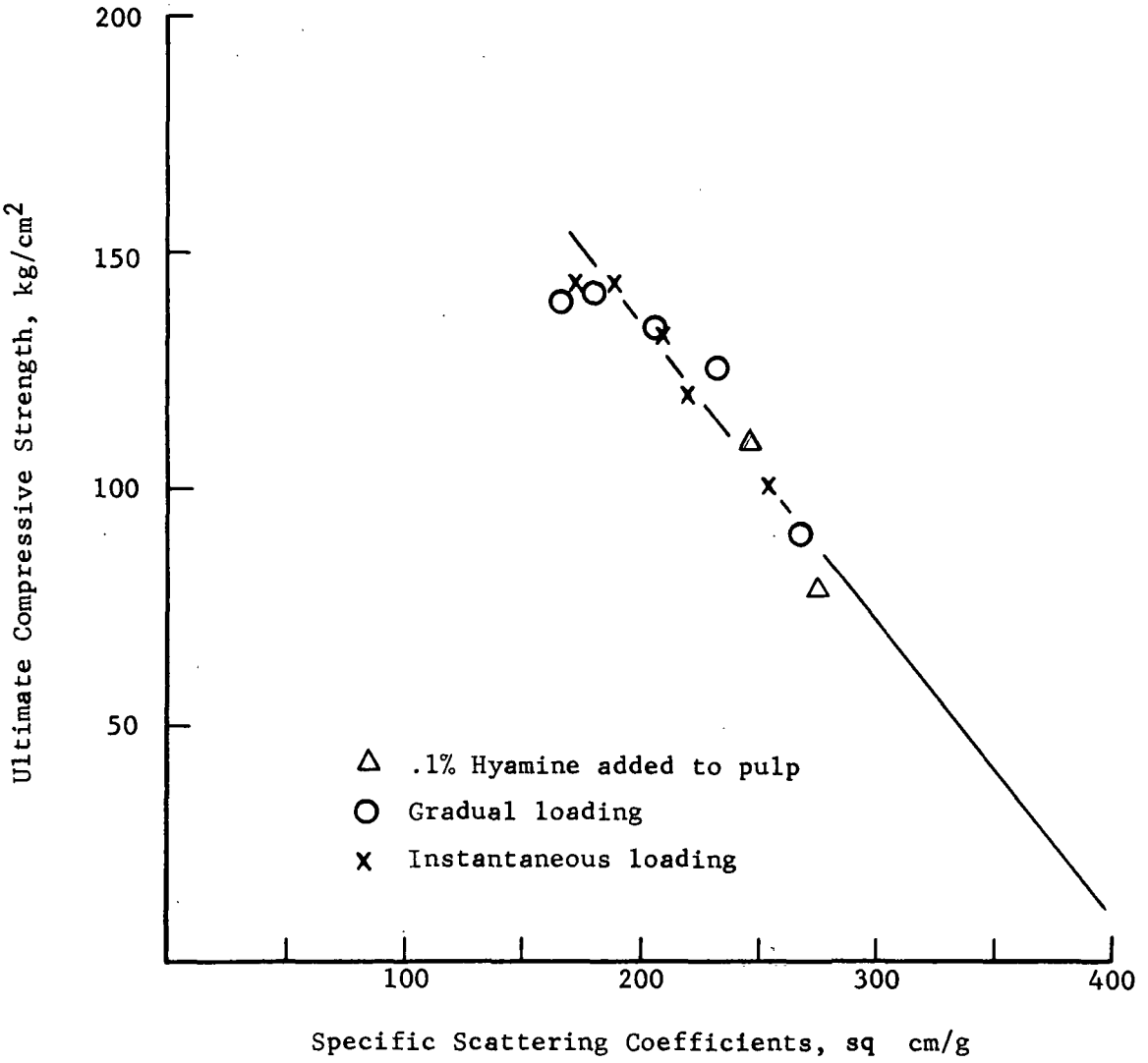

Figure 14. Relationship Between Compression Strength and Specific Scattering Coefficient (Northern Jack Pine Unbleached Kraft Pulp)

Preliminary results indicate that wet pressing might be used to adquately vary the sheet bond density at constant fiber stiffness. If this technique is used to produce the four sets of handsheets it will be necessary to more accurately determine the bonded area of the sheet and the fiber stiffness.

VARYING FIBER STIFFNESS

Very little attention has been devoted to a study of methods for varying fiber stiffness at constant bonding density. Accordingly, this phase of the preliminary study was directed at exploring a number of possible ways of varying fiber stiffness. Among these were the following:

- Acid hydrolysis of the fibers
- Exposure of the fibers to elevated temperature
- Radiation
- Use of melting agent

After considerable study exposure of the fiber to elevated temperature appeared to be the best trial method. This decision was prompted in large part by the results of a study carried out at The Institute of Paper Chemistry. In the reference study, loblolly pine chips were preextracted, delignified at 70°C using the chlorite process, and then extracted with caustic to remove the hemicelluloses. Portions of the pulp were then annealed by heating the fiber in water for at least two hours at 120 and 170°C under pressure.

X-ray diffractograms revealed a much higher level of order in the annealed fibers which indicated a greater degree of crystallization. The higher the annealing temperature, the greater an increase in crystallinity. Evaluation of the papermaking characteristics showed that annealed pulps hydrate more slowly during refining and that handsheets made from this pulp had lower tensile energy absorption and stretch but a 60-70% higher modulus; all are consistent with increased crystallinity. No measurement was made as to the individual fiber flexural stiffness; however, it would be expected that the increase in crystallinity would result in an increase in fiber stiffness.

In order to explore the feasibility of utilizing the annealing technique as a means of varying fiber stiffness, a quantity of the pulp delignfiied at 70°C was obtained. The pulp was prepared from loblolly pine chips. The delignification process — chlorite followed by caustic extraction — produces a bleached pulp; however, this was not considered a deterrent to its use in this phase since the objective is solely to determine the feasibility of varying fiber stiffness by heat annealing.

A portion of the pulp delignified at 70°C was heated at one of two temperature levels by placing its fibers (and water) in a pressure vessel which was submerged in an oil bath for two hours at either 120°C or 170°C. Three pulps — 70, 120 and 170°C — were refined to approximately 350 ml C.S.F. in a PFI mill, and then each was made into single and three-ply handsheets. For the purpose of determining the effect of wet pressing on annealed pulps, handsheets were made from each pulp at three different levels of wet pressing (Table V). The same sheet forming and drying procedures were used as described in the section entitled "Varying Bond Density."

TABLE V

PULP TREATMENT
(Loblolly Pine Bleached Pulp)

Sample Identity	Annealing Temperature, °C	Wet Pressing, psi
70/50	70	50
70/100	70	100
70/250	70	250
120/50	120	50
120/100	120	100
120/250	120	250
170/50	170	50
170/100	170	100
170/250	170	250

Handsheet Evaluation

The handsheets produced in this phase were conditioned and tested in the same manner and for the same properties as discussed in the section entitled "Varying Bond Density."

Discussion of Results

The objective of this phase of the preliminary study was to determine the feasibility of varying fiber strength by subjecting the fibers to a heat annealing process. The results of an unpublished recent Institute study indicated that heat annealing produced a significant increase in the crystallinity of the fibers. In the range of 70-170°C, the higher the temperature the greater the increase in crystallinity. Although no direct measurement has been made of the flexural stiffness of the heat annealed fibers, the flexural stiffness would be expected to increase with an increase in crystallinity. The trials carried out in this phase involved three levels of heat annealed pulps, each made into handsheets at three levels of wet pressing (Table VI).

The major objective in these trials is to determine if the annealing process increases the fiber stiffness. The primary property in this respect is the zero-span tensile test which was assumed to give an indirect measure of fiber stiffness. The zero-span results are retabulated below to more readily compare the effect at each wet pressing level.

Annealing Temp., °C	Zero-span Tensile Strength, km Wet Pressing Pressure		
	50 psi	100 psi	250 psi
70	12.8	12.6	13.3
120	12.7	13.3	12.5
170	13.0	13.9	13.3

TABLE VI

PHYSICAL PROPERTIES[a] OF HANDSHEETS
(Loblolly Pine Bleached Pulp @ 70°C
some annealed at 120°C and 170°C)

Sample Identification Number	Wet Pressing (psi)	Compressive Strength (kg/cm^2) (psi)	Basis Weight ($lb/M\ ft^2$)	Caliper (mil)	Scattering Coefficient After Compression (cm^2/g)	Relative Bonded Area (curve fit)	Fiber Strength Zero-Span Tensile (km)	Relative Bond Strength ZDT (ZDT/RBA) (psi)
70/50	50	158 (2253)	43.8	10.2	175.55	---	12.8	183 (---)
70/100	100	152 (2159)	43.6	9.8	147.74	---	12.6	216 (---)
70/250	250	157 (2228)	44.9	9.7	134.68	---	13.3	221 (---)
120/50	50	142 (2015)	40.0	9.2	186.44	---	12.7	211 (---)
120/100	100	133 (1885)	40.0	9.0	164.11	---	13.3	237 (---)
120/250	250	150 (2130)	41.9	9.2	137.78	---	12.5	244 (---)
170/50	50	162 (2302)	43.0	9.9	175.00	---	13.0	268 (---)
170/100	100	152 (2165)	45.1	10.1	153.69	---	13.9	264 (---)
170/250	250	150 (2131)	43.2	9.3	139.54	---	13.3	272 (---)

[a]All physical properties adjusted for basis weight except caliper.

The results indicate no clear cut trend for fiber strength increase with an increase in the annealing temperature. Only in the case of the handsheets wet pressed at 100 psi is there a progressive increase in zero-span tensile strength with increase in the annealing temperature. Although zero-span tensile is assumed to be an indirect measure of the flexural stiffness of the fibers, the lack of a definite increase in zero-span tensile with annealing temperature casts doubt on the validity of using zero-span tensile as a measure of fiber stiffness.

The ultimate compressive strengths of the handsheets are retabulated below.

Annealing Temp., °C	Compressive Strength, psi Wet Pressing Pressure		
	50 psi	100 psi	250 psi
70	2253	2159	2228
120	2015	1885	2130
170	2302	2165	2131

There is no dramatic change in compression strength with annealing temperature. In addition, the edgewise compression strengths did not increase with increasing wet pressing pressure. This appears to be due to the relatively high bonding strengths achieved with this pulp. The variations in wet pressing were included in this phase to see if the annealing introduced a significantly different effect compared to the behavior observed in the previous phase — "Varying Bond Density."

The scattering coefficients have been retabulated below for a more ready comparison:

Annealing Temp., °C	Scattering Coefficient, cm^2/g		
	50 psi	100 psi	250 psi
70	175.6	147.7	134.7
120	186.4	164.1	137.8
170	175.0	153.7	139.5

Although there is considerable variation in the data at the 50 and 100 psi wet pressing levels, the data clearly indicate that scattering coefficient decreases significantly as the total fiber-to-fiber contact area is increased by wet pressing. This confirms the results obtained in the previous phase.

The relative bond forces as determined by the z-direction tensile are also retabulated below:

Annealing Temp., °C	z-Direction Tensile Strength, psi		
	Wet Pressing Pressure		
	50 psi	100 psi	250 psi
70	183	216	211
120	211	236	244
170	268	264	272

Consideration of the bond force reveals some interesting trends. As would be expected, increasing the degree of wet pressing at each level of annealing tended to produce an increase in relative bond force. However, it was not anticipated that the bonding force at each level of wet pressing would significantly increase with increase in annealing temperature. The annealing process produced a greater change in relative bond force than the wet pressing. Increasing wet pressing increases bonding by creating a condition wherein more fibers or fiber

segments are brought into the field of molecular attraction or bonding. The increase in bonding force with increase in the degree of annealing at the same level of wet pressing indicates that the greater the degree of annealing, the more flexible and/or deformable the fibers become. This concept is, of course, contrary to the hypothesis based on selection of the annealing process as a means of increasing fiber strength. These results indicate that heat annealing may not be a feasible means of significantly varying fiber stiffness.

It may be noted that no values are tabulated in Table VI for relative bonded area. It may be recalled that relative bonded area is calculated from the following relationship:

$$RBA = \frac{S_t - S_u}{S_t}$$

where

S_t = specific scattering coefficient of the totally unbonded sheet

S_u = specific scattering coefficient of the handsheet

There are a number of ways in which S_t can be determined. In this study, the procedure used was one suggested by Thode and Ingmanson (14) which involves plotting the specific scattering coefficient versus ultimate compression strength and extrapolating ultimate compression to zero. The intercept (zero compressive strength) is an estimate of the scattering coefficient of a completely unbonded sheet. A plot of compression strength versus specific scattering coefficient for the annealed handsheets can be developed (Fig. 15). The data do not permit an extrapolation to zero compressive strength. Thus, relative bonded area could not be calculated by the method of Thode and Ingmanson. This method of determining the specific scattering coefficient of a totally unbonded sheet works reasonably well with the unbleached pulp used in the previous phase, "Varying Bond Density."

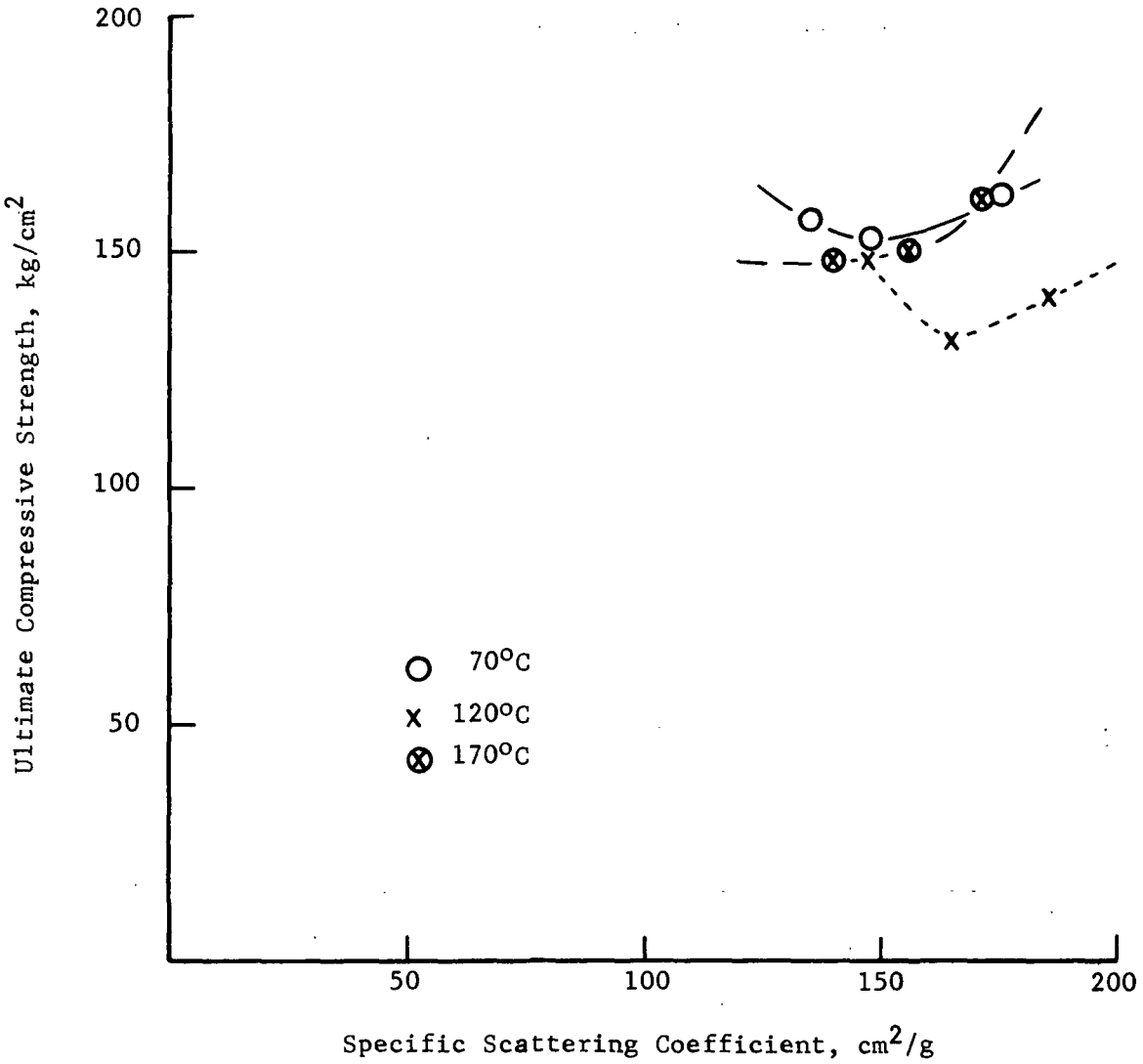

Figure 15. Relationship Between Compression Strength and Specific Scattering Coefficient (Loblolly Pine Bleached Kraft Pulp)

The chlorite delignification process used in this phase resulted in a bleached pulp. Swanson and Steber (18) have reported that the extrapolation technique may not be adequate in the case of purified pulps. This anomaly may possibly be due to the fact that the optical system may not be absolutely correct for both bleached and unbleached pulps. The optical measurements reported in this phase ("Varying Fiber Stiffness") were made at 1150 nm wavelength. A trial was made in which reflectance and transmission curves were obtained for bleached and unbleached handsheets (at equal basis weight) over the wavelength range of 1000-2500 nm. This trial was carried out to see if there was a wavelength which was independent of the degree of purity of the pulp. The results (Fig. 16) indicated that 1150 nm was in the area of minimum purity effect on reflectance, $\underline{R}_o$. There was no common wavelength for transmittance.

The validity of the assumption that the zero-span tensile strength is an indirect measure of fiber stiffness is in serious doubt. An analysis of handsheet properties (Table VI) shows no increase in zero-span tensile with an increase in annealing temperatures. However, results of a study carried out at The Institute of Paper Chemistry shows a 60-70% increase in the tensile modulus of sheets made from annealed fibers. Annealing increases the crystallinity of the fiber which should result in an increase in fiber stiffness; thus, if zero-span tensile is a valid indirect measure of stiffness, the results do not indicate it.

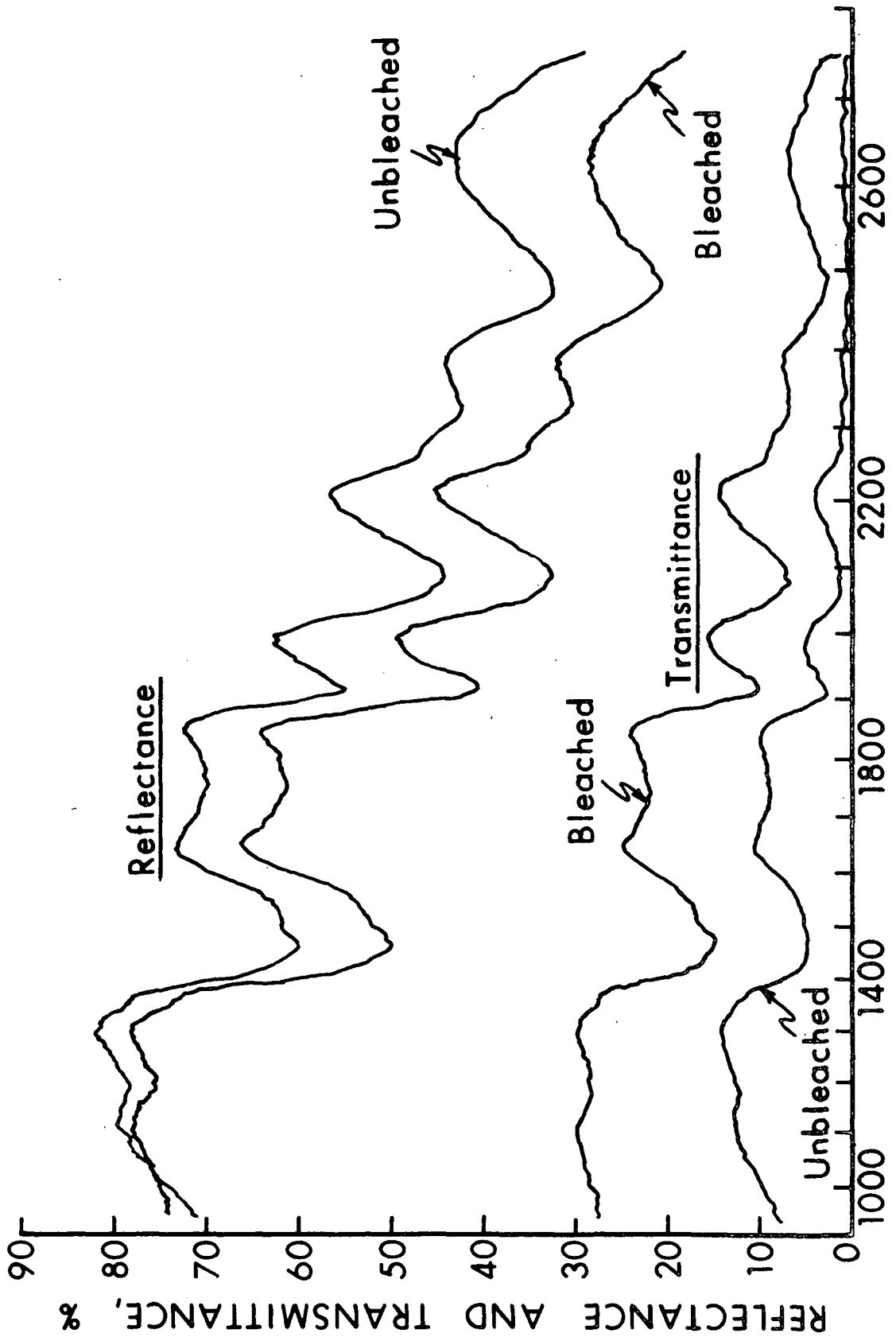

Figure 16. Transmittance and Reflectance Curves, Bleached and Unbleached Samples

CONCLUDING REMARKS

The task of developing the four sets of handsheets still remains. If the work completed to date is essentially correct (Fig. 11), a wet pressing pressure between 50 and 300 psi may be adequate to produce the desired variation in bond density. The task of varying fiber stiffness is more difficult than varying bond density. Exposing the fibers to an elevated temperature may be a valid approach. If not, other methods, such as choosing springwood and summerwood, can be explored. Regardless of the methods used to vary fiber stiffness and bonding, adequate procedures for measuring the fiber stiffness must be employed.

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